



# Structures and properties of layered bioceramic coatings on pure titanium using a hybrid technique of sandblasting and micro-arc oxidation



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## ABSTRACT

Sandblasting is the most ordinary approach not only to leave the treated regions in compressive residual stress states but also to alter the surface topography of an implant, and micro-arc oxidation (MAO) provides a novelly effective way to produce porous, adhesive and bioactive implant coatings. In this study, ceramic coatings containing Ca and P elements were deposited on the sandblasted pure titanium substrates through the MAO process, and the bioactive performance of the coatings was improved. In addition, the variation of morphology and microstructure, phase and element composition of the coatings according to treating time and related properties were characterized and analyzed, respectively. It was indicated that the hybrid-treated coatings exhibited better properties than that by MAO method, especially in hydroxyapatite (HA) inducing ability, as evidenced by characterization test and HA formation after simulated body fluid (SBF) immersion for days. The enhancement of modified surface was attributed to the combination of the physical and electrochemical treatments.

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## 1. Introduction

Titanium (Ti) and its alloys have excellent biocompatibility, corrosion resistance, mechanical properties, and light weight [1]. They have become the most striking metallic materials for the purpose of orthopedic implants in recent years [2]. In ensuring their long-term osseointegration, the surface properties of Ti implants play a key role [3,4], and there have been many topological and chemical modification techniques to enhance osteoinduction on the surfaces [5].

In dental area, a roughened surface of Ti and its alloys was shown to markedly increase osseointegration, cellular differentiation and mechanical fixation [6,7]. To create the appropriate value of roughness, several techniques were developed, including sandblasting, acid etching, SLA (sandblasting, large-grit, acid-etching) and so on [8]. Sandblasting is a primary technique to modify the mechanical properties of the metal surface and near surface region through the induced severe plastic deformation, leaving the treated regions in compressive residual stress states [9]. Although it could create a coarse surface to facilitate the adhesion of cells and the growth of

new bone, the surface roughness is not completely identical and uniform in the whole [10]. On the other hand, grit-blasting materials are often embedded into the sandblasted surface and residue remains after ultrasonic cleaning and acid passivation, potentially hindering the osseointegration process [11]. On its own, the sandblasting method may not be effective enough to achieve early fixation and bone formation. Subsequently, additional surface modification of implants may be needed when the bone quality is poor [12–14].

Another strategy to improve osseointegration is to coat the implant surface with bioactive components [15]. Compared with other modifications for producing bioactive coatings, micro-arc oxidation (MAO) has been one of the most applicable methods to deposit bioceramic layers on implants and provide the possibility for incorporating Ca and P, which can further crystallize into hydroxyapatite (HA) or other calcium phosphate [16–18]. Moreover, it is noteworthy that MAO can form coatings on a geometrically complex surface, and this is of importance for enabling the bone-bonding ability of implants [19–21]. However, to achieve a bioactive coating with high-quality performance, this technique requires further development.

In this study, we examined titanium oxide coatings formed by sandblasting and MAO with a mixture of Ca- and P-containing system. As most literatures reported producing a bioactive coating by

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**Table 1**  
Process conditions for the micro-arc oxidation treatment.

Electrical parameters (positive/negative)			Electrolyte concentration (g/l)			System temperature (°C)	Treating time (min)
Voltage (V)	Frequency (Hz)	Duty cycle (%)	(CH <sub>3</sub> COO) <sub>2</sub> Ca·H <sub>2</sub> O	Na <sub>2</sub> HPO <sub>4</sub> ·12H <sub>2</sub> O	NaOH		
450/0	600/0	20/0	5.285	2.148	0.250	≤40	10, 15, 20, 25, 30

**Table 2**  
Process conditions for the sandblasting treatment.

Abrasive size (μm)	Air pressure (bar)	Flow rate (m/min)	Impact angle (°)	Nozzle distance (cm)	Treating time (s)
SiC 180	8	2.17	90	10	30

sole MAO technique, the advantageous properties of the coating were limited. The present study aimed at forming a bioceramic MAO coating on the sandblasting treated pure titanium and having, thus, synergistic effects of the modified substrate with an oxide layer. These coatings were fabricated by hybrid treatment, which not only determined their original morphology, but also possibly modified surface chemistry as well as characteristics due to the combination of mechanical enhancement and electrochemical oxidation.

## 2. Experimental

### 2.1. Sample preparation

Commercial pure TA2 Ti discs with a diameter of 14 mm and thickness of 2 mm were used as the substrates. The discs were superficially polished with 180–800# abrasive papers to a roughness of  $R_a = 0.2 \mu\text{m}$ , ultrasonically cleaned in acetone and distilled water, and then dried in air.

### 2.2. MAO treatment

The MAO treatment was carried out in a water-cooled bath made of stainless steel, which served as a cathodic electrode, and the samples prepared were anodized for 10–30 min in accordance with detailed processing conditions in Table 1. Bipolar electric rectangular pulses were applied to the specimens, fed from a 45 kW AC-type power supply. The micro-arc oxidation device provided a preset constant voltage, and the current varied with the duration of anodizing time. In the experiments, aqueous solutions of electrolytes were prepared to mainly contain (CH<sub>3</sub>COO)<sub>2</sub>Ca·H<sub>2</sub>O and Na<sub>2</sub>HPO<sub>4</sub>·12H<sub>2</sub>O, with a Ca/P ratio of 5:1. A controlled amount of NaOH was also added in to adjust its alkalinity and increase conductivity. The electrolyte was cooled by a cooling system to keep its temperature below 40 °C. After the treatment, the samples were washed with distilled water and dried in an air oven. For comparison and analysis, the samples treated for 10, 15, 20, 25, 30 min were labeled as the MAO-treated group (M-group: M-T10, M-T15, M-T20, M-T25, M-T30) for testing.

### 2.3. Hybrid treatment

The hybrid treatment involved two steps. First, the polished discs were sandblasted by applying a jet of SiC particles in tiny size at a high pressure until the surface reached a uniform gray tone. The technical factors that could influence the sample's properties were: abrasive size, compressed air pressure, mass flow rate, impact angle, process time and distance from the nozzle to samples, as shown in Table 2. After sandblasting, the pretreated samples were anodized for 10–30 min by MAO technique using the same treatment conditions as listed in Table 1 to accomplish the hybrid

treatment. Afterwards, the coated samples were also flushed, dried, and labeled as the hybrid-treated group (H-group: H-T10, H-T15, H-T20, H-T25, H-T30) according to the varied treating time of 10, 15, 20, 25 and 30 min.

### 2.4. Immersion in SBF

The simulated body fluid (SBF) was prepared by dissolving the reagent-grade chemicals into distilled water in order as listed in Table 3, and buffering at pH 7.40 with Tris (hydroxymethyl) aminomethane and dilute HCl at 37 °C. The ionic concentrations of SBF were also listed in Table 4. Both the M-group and H-group specimens were soaked in 20 ml SBF for 5 and 7 days for comparison, and the SBF was refreshed every day.

### 2.5. Characterization

The morphology was examined by SU-70 field emission SEM (FE-SEM). Because of the low conductivity, the sample was sputter-coated with Pt prior to SEM. The thickness was measured by Mini Test 600B FN2 thickness meter. The phase analysis of the coatings was carried out using Rigaku D/max-γB X-ray diffractometer (XRD), with a scan speed of 4°/min, operated at 40 kV and 100 mA. JXA-8800R electron probe microanalyser (EMPA) with a Link ISIS300 energy spectrum analyzer was used to give the secondary electron (SE) image and the element composition analysis was conducted by energy dispersed spectroscopy (EDS). Microhardness and scratch tests were carried out to evaluate mechanical performance of the

**Table 3**  
Amounts of reagents for the preparation of SBF.

Order	Reagent	Purity	Amount
1	NaCl	99.5	8.035 g
2	NaHCO <sub>3</sub>	99.5	0.355 g
3	KCl	99.5	0.225 g
4	K <sub>2</sub> HPO <sub>4</sub> ·3H <sub>2</sub> O	99.0	0.231 g
5	MgCl <sub>2</sub> ·6H <sub>2</sub> O	98.0	0.311 g
6	1.0 M-HCl	–	39 ml
7	CaCl <sub>2</sub>	95.0	0.292 g
8	Na <sub>2</sub> SO <sub>4</sub>	99.0	0.072 g

**Table 4**  
Ionic concentrations of SBF.

Ion	Concentration (mmol/l)
Na <sup>+</sup>	142.0
K <sup>+</sup>	5.0
Mg <sup>2+</sup>	1.5
Ca <sup>2+</sup>	2.5
Cl <sup>-</sup>	147.8
(HCO <sub>3</sub> ) <sup>-</sup>	4.2
(HPO <sub>4</sub> ) <sup>2-</sup>	1.0
(SO <sub>4</sub> ) <sup>2-</sup>	0.5

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